

Hi, my name is Stephanie Benight, and I work for Larry Dalton at the University of Washington, in Seattle, at the chemistry department. Today I'm going to show you the technique called variable angle spectroscopic ellipsometry or VASE. Several different properties can be measured using variable angle spectroscopic ellipsometry. Most importantly we can ascertain film thicknesses of samples, refractive indices, surface roughness, interfacial mixing, composition, crystallinity, anisotropy, and also uniformity of different materials.

So in our research as part of the STC has been to this technique to ascertain refractive indices of different electro-optic chromophore materials. Here in this graphic we show that have we have light incidental on the surface of our substrate or our sample. And this shows the different polarizations of light from the source that are being reflected off of the sample.

And so what ellipsometry does is it detects the changes in the polarization of the light that's reflected off the surface in both the magnitude of the polarization, and the changes in the phase of the polarizations of light.

The light that's being detected is expressed in terms of coefficients Psi and Delta and those are representative of both the magnitude and the phase.

Other components of this that are also detected are coefficients N, C and S and these carry the information on the changes in intensity.

So this is the ellipsometer. And when we're conducting a measurement we have light that comes through from this arm here and it's important to note that this arm as well as the arm of the detector can move at different angles.

And so when we are conducting a measurement we have input light from this arm coming in and is able to shine on our sample which is mounted on our sample stage here. It's also important to note that the sample stage can move horizontal in both directions, and there's also as a tilt that's associated with it to make sure that the light is incident level on our sample.

And so what we're measuring with ellipsometry is we have incident polarizations of light that hit the sample, and the sample is going to alter those polarizations of light and then send different information about changes in the light with respect to the magnitude and the phase of the light into the detector.

So when we conduct a measurement we want to use multiple angles of incidence when we collect our measurements. And these arms are mechanical and they move up and down depending on the manually inputted angles of incidence that we want.

So a variety of different types of materials can be measured using the VASE technique such as transparent materials like a glass slide, absorbing materials- here is a dye as thin film on glass- and then also opaque a substrates such silicon oxide on silicon as shown here, also different metal layers on substrates, and transparent conductive oxides like ITO can also be measured using this technique.

So now we're going to through an example and what we're going to do is measure a glass slide.

And so the first thing we need to do- we have a glass slide here – is to simplify our data analysis later on is what we can do is apply a piece of Scotch tape to the back of our substrate, because when measuring transparent substrates there can be backside reflection that we will have to take into account in our model later on, and so this can simplify things if we do this right away.

The next thing we'll do is mount our slide onto the sample stage, and we also can adhere a vacuum if we choose but since we have a relatively big sample we really don't need that -the sample will lay evenly on the stage.

And so once we have our sample prepared and mounted the first thing we should do align sample and we want to make sure that our incident light is reflecting off of the sample into the detector.

So I'm going to look inside the detector, and inside there's a small ring in which the light is being fed into, and so we can read the information in the software.

When doing the sample alignment were trying to move the collected light into the aperture so we have an adequate amount of light being collected that's reflected off of the surface of the sample.

Another way we can tell that we have an aligned sample is that we see this saturation here into the detector as indicated in these dark gray signals.

If we move it out enough we no longer have adequate collection of light into the detector. This is also evident by looking at the intensity, if we move the light back into the detector we can see the intensity also start to climb and we see appearance of the gray saturation indicating that we're going to have enough light collected to collect adequate data.

So now we will manually input parameters to set up for measurement. It's important to note that the software is unique to this instrument manufactured by the J. Woollam Company. Your instrument that you are using may have different software that's used.

The first thing we're going to do is set up our measurement. In doing so will first have to assign a file name to our sample, how about "glass test", OK, save. And this allows us to input our parameters for the measurement. And so we want a standard measurement with an acquisition time of four seconds. And this is where we can assign our angles of incidence and so what we're going to do is scan at 55 degrees to 75 degrees, increasing by 10 degrees.

So the rest we have an automatic alignment, we also would want an automatic quick sample height alignment. It's important approximate our sample thickness and since we are measuring a standard glass slide we're going to start at 1 millimeter.

At this point we can start by measurement, and click OK.

So now we can see that when you see the gray exposure here that means that light is hitting the detector this is measuring the intensity, and now we're acquiring our data at 55 degrees.

So now it's acquiring data at all the different wavelengths simultaneously from 280 nanometers to 1700 nanometers in the case of this instrument. And now it's moved to 65 degrees and now it's moving to our final angle of 75 degrees.

And what we have in our output now that the measurement is done is our Psi and Delta data as shown here, and the data for angles of incidence at 55 degrees, 65 degrees, and 75 five degrees are shown.

So here's the result of our measurement and we have both Psi and Delta data shown on the screen.

The Psi data is given in red, while the Delta data is given in green. It's also possible to view these only looking at just the Psi data or just the Delta data.

And the axes that we're looking at- the magnitude of Psi coefficient is on the left with respect to wavelength on the X axis, and the axis on the right is Delta.

So in acquiring useful data like the film thickness and refractive index of our material we've collected our optical data Psi and Delta, and different constants and now what we need to do is build a model to fit our experimental data and analyze our model.

And what we do is use a regression analysis to fit different parameters in our model to our experimental data.

And so the first thing we need to do is build a model.

And so if we go- we have our data pulled up here- we go in the analysis tab at least in this software we can open a model that we want to start with.

In the instrument there are several different models built-in and here we'll start with a glass substrate since that's what we've measured, and that gives us the starting point for us to build the model to adequately fit the data from our glass sample.

So the first thing we need to do is maximize our substrate and see all of these parameters inside this box. In our example since we have a glass slide which is a transparent substrate we want to use what's called a Cauchy model of dispersion to model this sample. And so with that A is basically the refractive index of the substrate where as B and C actually model the behavior of the dispersion of the sample.

And so in order to fit our data, if we click "generate", that shows us in the black dotted line where our model is currently fitting.

And you can see that in the UV in wavelength range here around 300-500 nanometers the model doesn't fit too well.

So what we can do is assign different values as initial guesses to try to fit the data.

And so here we have zero as a starting point of the first way to fit the dispersion.

What we can do is assign this a specific number, in our case for glass .02 is a good estimate. And so when you assigned numbers it automatically switched to our refractive indices of both N and K, where N is the real portion of the refractive index, and K in green is the imaginary portion.

In the model we're using here you can only use it with transparent substrates in which K or the absorption of the material is very minimal or almost zero.

And so if we go back to our Psi and Delta data for now to make things simple I'm going to right-click on the C parameter and only fit the A and B parameters. So if we generate our model and click "fit" then the software does a regression analysis to fit our data and from that since parameters of A, B, and surface roughness are in bold they were altered in the fitting and regression analysis to try to fit the model we had generated to our experimental data.

If we look at our Psi and Delta data with fit in the black line we see then the model very nicely fits our data.

Another way to look at this is to see that the mean squared error of our fit given in the left hand corner here is very low. We have a MSE value of 3.981 which is very low indicating we have a nearly perfect fit of generated model to our experimental data.

So now have a generated model we see they we're still looking at measurements of different changes in magnitude and phase of the polarization. To actually look at the refractive index of the material we want to right-click on our substrate and graph the layer optical constants.

And so now we have a graph of N and K of our material for a variety of different wavelengths which we scanned over.

And specifically N here is shown in red and that's the real portion of the refractive index of the glass substrate, and K is shown in green which is nearly zero.

Furthermore the N value is shown on the left hand side axis and K values are shown on the right hand side, all as a function of wavelength shown on the X axis here in nanometers.

So the more noisy signal here it's important to note is not indicative of the material that was measured but rather of the instrument that's being used. As we see here when we're looking at the data that we do intend on measuring we have our optical constants and you can see you then in IR region that noise does not translate to our generated model.

Now we will repeat our measurements with a strongly absorbing sample.

So now we've acquired data for an absorbing film on glass we have our Psi and Delta data still at three angles of incidence; 55 degrees, 65 degrees, and 75 degrees shown here. And as you can see the Psi and Delta data appears quite differently for an absorptive film compared to just a transparent material like glass that we saw previously.

If we wanted to build a model to fit this data the problem becomes a little more complex. But initially, since this is an absorptive film on glass, what we would do is build the model for both our glass substrate and our organic layer on top.

And so we would begin as we did in our previous example with a glass substrate. If we want to fit just our top layer we need to add a layer that is only for that layer of material.

And so what we can do is add a layer on top, and for our purposes here we'll start with a different type of material. We know that we use a Cauchy model for transparent materials, these other models can be used for absorptive materials. For our case we will use what's called a B-spline model, and that will be representative of our material of interest, our absorbing film that we just measured.

So before we model the absorptive film we first want to maintain our optical constants for our glass because that didn't change in our new measurement. Upon starting to model our material layer we want to start with what we think would be an approximate thickness.

And in this case I think the thickness is around 400 nanometers, and that might be a good starting point. I'm also going to set the surface roughness to zero and turn that off entirely and make our fit appear much better.

So now what we would do is go through, generate a model, fit the model, and then tweak the parameters until we obtain a model that we're happy with that successfully models our material.

Here we have an example of a model fit to a strongly absorbing film and you can see that we have our glass substrate as part of our model here, and we have a layered designated to our absorbing film here.

After toggling with different parameters we're able to achieve a fit, again shown in the diamond black line, that's reasonably correlated with our experimental data. And another way to test that is to look at our MSE value and see if it's at least below 100- ideally around 20. The lower the MSE value the better the linear regression analysis and the better the fit we have.

And now we've fit a good model to our Psi and Delta data it's important to look at what we're interested in which is our refractive indices of the material versus wavelength. So if we right-click on our model, and graph our optical constants, here on the left axis we have the magnitude of N or the real component of our refractive index. And on the right hand side axis we have the magnitude of K or the imaginary or absorptive component of our refractive index versus wavelength which is on the X axis.

So in this case for this sample we see that looking at the green curve that represents the absorptive component of refractive index we have a large peak at around 800 nanometers, and from the experiments that I've done for this sample it correlates quite nicely to the absorption that we detect using a method like UV visible spectroscopy.